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SELECTED PLASMA NITRIDING METHODS USABLE FOR THE THERMO-CHEMICAL TREATMENT OF AIRCRAFT PARTS

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Abstract

The aim of the work was to review selected methods of plasma nitriding, which according to the authors might be used in the thermo-chemical treatment of aircraft parts. The introduction explains the nitriding process and presents the requirements on the thermo-chemical treatment of aircraft parts. Three methods of plasma nitriding have been described: Direct Current Plasma Nitriding (DCPN), Active Screen Plasma Nitriding (ASPN) and Low Pressure Nitriding in AEGD (Arc Enhanced Glow Discharge) plasma. While describing DCPN plasma nitriding method, authors drew attention to known problems, which occur in this process such as edge effect or hollow cathode effect. The Keller's model, the Marchand's model and the Walkowicz's model of nitriding process, which can be found in the literature, were also presented in this work. Another purpose of this work was to present hypotheses about the transportation of nitrogen during Active Screen Plasma Nitriding process and to show that in this nitriding method, defects typical for Direct Current Plasma Nitriding do not occur. While describing Low Pressure Nitriding in AEGD plasma, authors also presented the model of nitriding mechanism in this process, which shows four nitrogen diffusion paths (physisorption, chemisorption, adsorption of N_2, ion implantation). Examples of layers obtained by applying described nitriding methods were presented. The measurement of nitrided layers confirmed that it is possible to obtain a layer without ε film on the surface.

Keywords: *plasma nitriding, aircraft part, Direct Current Plasma Nitriding, Low Pressure Nitriding in AEGD (Arc Enhanced Glow Discharge) plasma, Active Screen Plasma Nitriding*

1. Introduction

Modern aircraft parts should be characterized by such features as high surface hardness, high resistance to abrasive wear, high resistance to fatigue and corrosion [1, 2]. Among many techniques which have an influence on the surface properties, such as forging, casting or machining, both non-diffusion heat treatment (quenching) and a diffusion one (carburizing, nitriding) have been also developed. Nitriding consists in diffusive saturation of the surface layer of metal elements made of steel, cast steel and cast iron with nitrogen [5, 6]. Nitriding of the surface layer of machine parts made of steel allows obtaining high hardness, high abrasion resistance, corrosion resistance and fatiguing strength of the workpieces. Thanks to so many advantages of this, process several variations, including: gas nitriding, nitriding in fluidized beds, powder nitriding, salt bath nitriding and plasma nitriding have been worked out. At present, the most promising, also for applications in the aerospace industry, are the methods of plasma nitriding and the greatest emphasis is put on their development [3, 5-7].

The structure of the nitrided layer created on a thermo-chemically improved element depends mainly on the temperature of the process and activity of nitrogen. The phase composition of nitrided layers produced on iron is always formed according to the scheme α – γ' – ε. At the beginning the layer of unsaturated solution of α-Fe(N) is formed, which begins to crystallize in the form of iron nitride γ'-Fe_4N due to supersaturation of its surface layer. Further diffusion of nitrogen in the phase γ' leads to the increase of its thickness in addition, it moves the boundaries of α and γ' phases deep into the material. After saturation of γ' layer, on its surface nuclei of hexagonal phase ε-$Fe_{2\text{-}3}N$ are formed, which are stable only at higher concentrations of nitrogen. On cooling, these layers have a tendency to break down into two-phase areas of α + γ', and ε + γ' [4, 7].

Currently, the aerospace industry requires production of repeatable nitrided layers consisting of several-micrometre thick iron nitride layer and adjustable diffusion layer of internal nitriding, which requires strict control of processing parameters of the carried out thermo-chemical processes. Such nitrided layers have good formability behaviour and maintain very strict dimensional tolerances [1, 2].

2. Direct Current Plasma Nitriding (DCPN)

Direct Current Plasma Nitriding (DCPN) is one of the most widely used technologies of surface treatment of metals. In the standard diode configuration of DCPN method, nitrided elements are placed on a conductive metal table inside the vacuum chamber. The table and nitrided elements located on it, are connected to the high cathode potential and the metal walls of the vacuum chamber perform the role of the anode. Such a high cathodic potential is responsible not only for the creation of an abnormal glow discharge on the nitrided substrates, but also for their heating in result of intensive bombardment with fast ions and energetic neutral particles of the reaction gas. The type of active particles generated in the plasma and the way they interact with the substrate surface are strongly dependent on the composition and the pressure of the process atmosphere, the discharge voltage and the type of substrate material. DCPN processes are carried out at the atmosphere pressure range from 1 to 5 mbar in so-called dynamic vacuum. In the process, by means of special multi-channel dosing systems, gas mixture is supplied to the chamber depending on the needs and conditions of the process. Diagram of the device used for DCPN technology is shown in Fig. 1 [3-7].

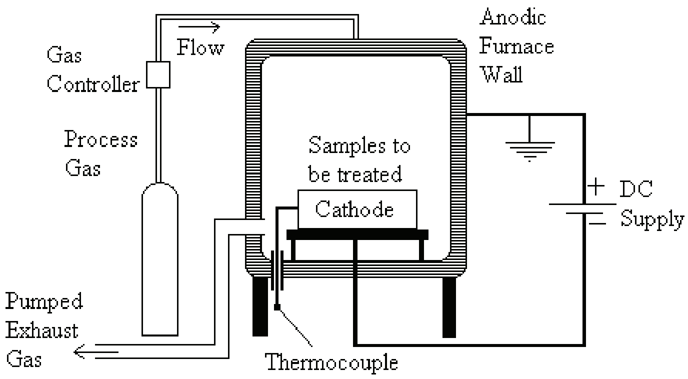


Fig. 1. Diagram of the device used for DCPN method [3]

After evacuation to the required initial pressure, the vacuum chamber is filled with the gaseous mixture to the correct process pressure, by means of flowmeters. After switching on the power supply, in the chamber the glow discharge is initiated, in which the active particles of nitrogen (e.g. in the form of positive ions N^+, N_2^+) are created and accelerated towards the negatively polarized elements placed on the worktable. During bombardment of the surfaces of the elements, active particles transmit kinetic energy and heat them so intensely that it is unnecessary to use additional heating systems [3-7].

With the change of the researchers' views on the role of the main elements of the gas-plasma environment responsible for nitrogen diffusion into the substrate, the models describing the mass transport during plasma nitriding in a DC diode glow discharge have changed as well. The Keller's model of 1971 was based on the hypothesis that the process of nitrided layer growth is entirely determined by the reactive sputtering of the substrate surface (Fig. 2a). In the Marchand's model proposed in 1990, contradictory to the Keller's one, the leading role in the nitriding process was assigned to the neutral particles N and N_2. Sputtering of the cathode surface impedes nitriding process here (Fig. 2b). The model developed by J. Walkowicz in 2003 takes into account the role of both, ionized particles and neutral particles in the nitriding process (Fig. 2c). Research on the plasma structure also allowed designating a quantitative share of these particles in the plasma discharge [4-9].

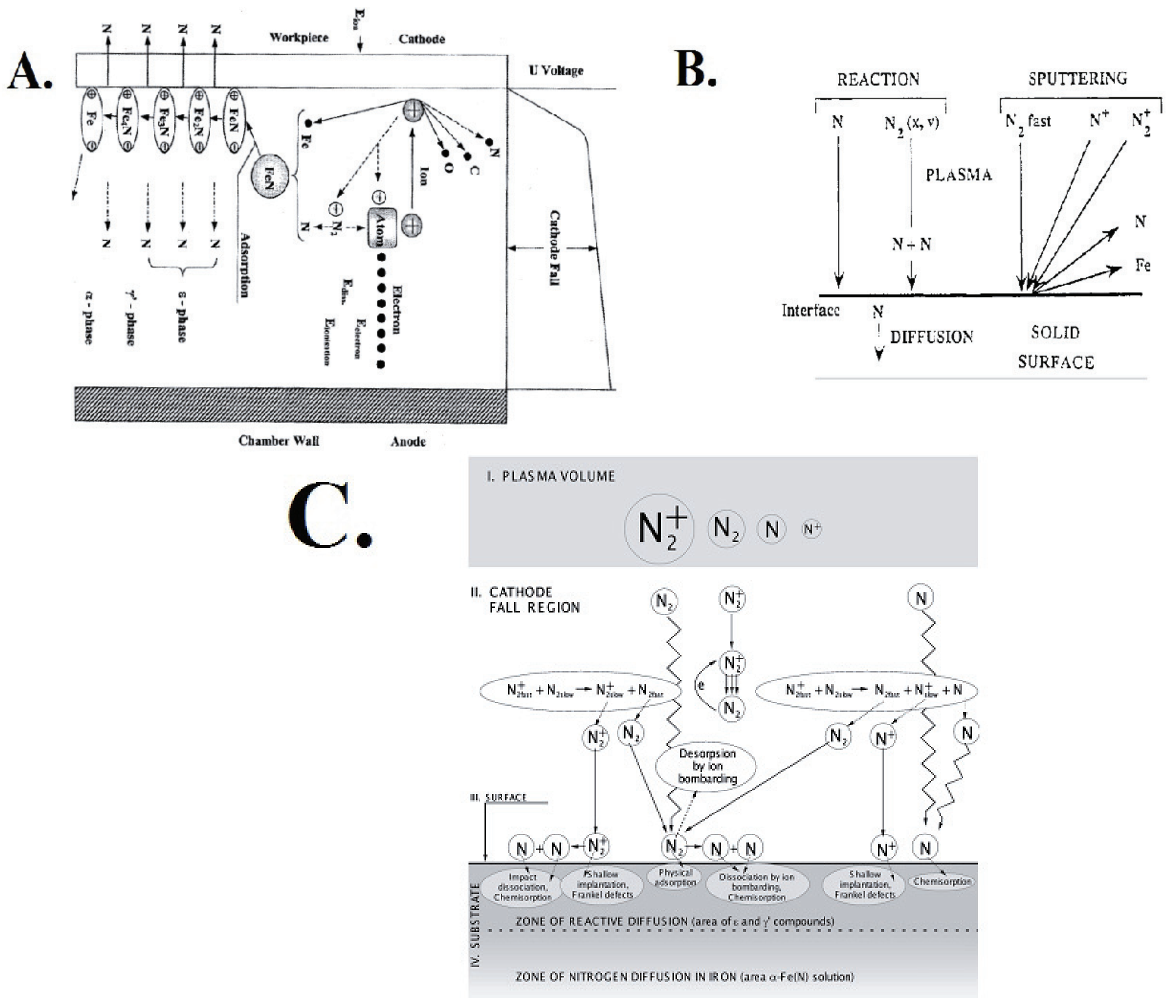


Fig. 2. The evolution of plasma nitriding model in a diode configuration: A. Keller's model [16], B. Marchand's model [16], C. Walkowicz's model [8]

Nitriding method in DCPN system has many disadvantages, apart from some advantages, such as the edge effect manifested with overnitriding of sharp edges caused by non-uniform electric field distribution, the hollow cathode effect that occurs when the nitrided element has a cavity in which the reduced distance between the planes of the cathode causes interference of two separated discharges. These defects can cause problems when using this method in thermo-chemical machining of aerospace components.

Figure 3 shows a metallographic section of nitrided steel PN 16HG (EN 16MnCr5) sample. The item was nitrided by DCPN method. In the picture, one can see that there is no metallurgical zone of hard nitrides ε. The nitriding process in DCPN configuration was controlled to the degree that it was possible to obtain nitrided layers without ε zone, which is important for thermo-chemical processing of aircraft parts.

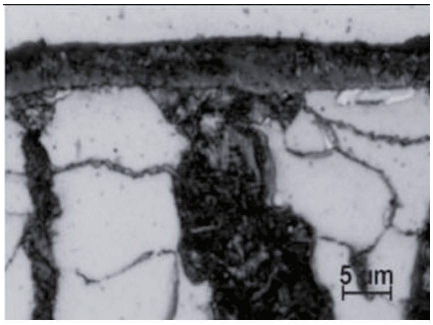


Fig. 3. Metallographic section of nitrided steel PN 16HG (EN 16MnCr5) sample [6]

3. Low Pressure Nitriding in AEGD (Arc Enhanced Glow Discharge) plasma

Arc Enhanced Glow Discharge (AEGD) consists of glow discharge caused by electrons emitted from the vacuum arc. For this purpose, the arc sources intended to deposit thin films of a vacuum arc evaporation method are used. Electrons emitted by a working arc source are accelerated by an auxiliary anode. During the flow from the cathode to the anode, they ionize the working gas, which is under reduced pressure. The scheme of the method used for nitriding in AEGD discharge is shown in Fig. 4 [10, 11].

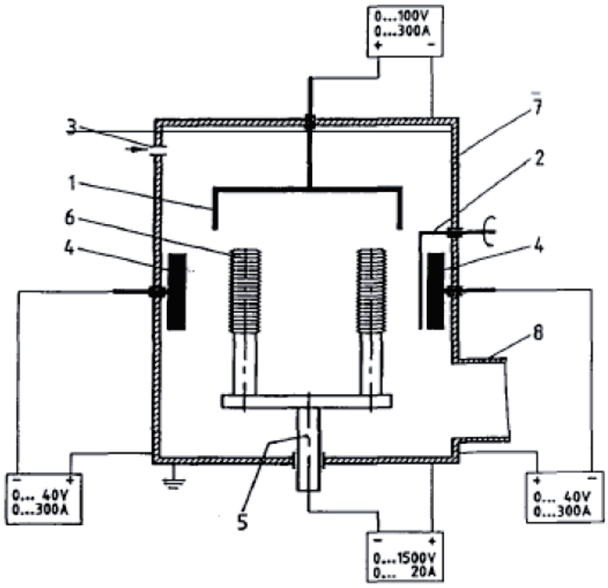


Fig. 4. Scheme of the implementation of the AEGD discharge [11]. 1- separate anode, 2- shutter, 3- gas inlet system, 4- cathodes, 5- substrate holder, 6- nitrided elements, 7- vacuum chamber, 8- pumping system

To separate the workspace from a metal vapour, the arc source discharge remains shielded during AEGD nitriding. The positive polarity of the substrates, in relation to the grounded chamber walls, allows for its use as an additional anode. This leads to intense bombardment by electrons and consequently to its heating. This solution has many advantages [10], which include:

- the energy of the electrons is lost only on positively polarized components. Thus, the heating is selective and restricts the desorption of gases from other surfaces,
- a stream of atoms and ions of metal (usually titanium) emitted from the cathode arc acts as a getter for the gases desorbed from the elements of the technological system,
- heating by the electrons does not result in etching of the workpieces.

Due to the arc current of 60 to 300 A, a high concentration of electrons is achieved in the AEGD discharge. The potential of the anode responsible for the acceleration of electrons, measured in relation to the ground, reaches 100 V. Studies on the AEGD discharge characteristics carried out by Vetter [10, 11], show an increase in the electron current at the auxiliary anode with the increasing anode bias. This allows for continuous adjustment of the discharge intensity, and allows controlling the process of the workpiece heating [10].

The nitriding mechanism in AEGD method is not clearly established. However, four main roads leading to the diffusion of nitrogen are indicated [10, 12].

Physical and chemical adsorption of nitrogen molecules and nitrogen atoms take place on the surface regardless of its polarity. The adsorbed nitrogen molecules N_2 under the influence of high temperature and excited by bombarding electrons are dissociated to form a layer of atomic nitrogen on the surface. Atomic nitrogen dissociated in the workspace volume has also its participation in this layer. In the last stage, the nitrogen diffuses into the material. In the case of a negative polarity of the substrates, adsorption of atomic and molecular nitrogen is limited. Ions accelerated by electric field knock out the nitrogen adsorbed on the substrate surface. As a result of the bombardment, a fewer number of nitrogen atoms is diffused. If the workpiece is negatively biased, the occurrence of shallow ion implantation is possible. It occurs when the ion energy of the nitrogen is sufficient to penetrate the crystal lattice of steel. Fig. 5 shows a model of mass transfer in AEGD nitriding [10, 12].

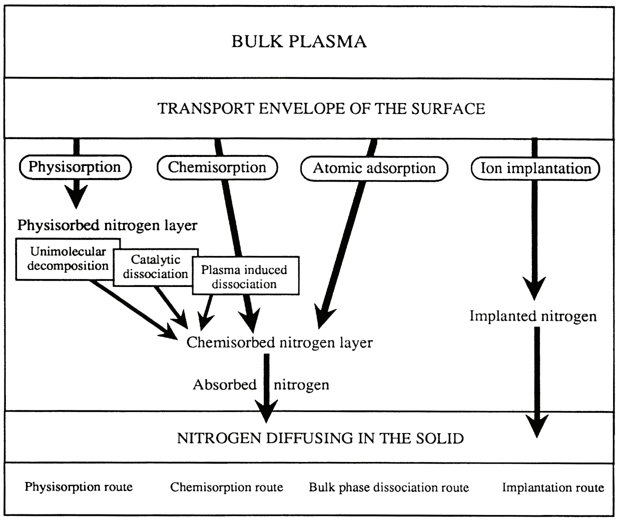


Fig. 5. A mass transfer model in AEGD nitriding [12]

Figure 6 shows a metallographic section of the etched high speed steel SW7M sample at which no zone of hard nitrides ε was formed. The item was nitrided using Low Pressure Nitriding in AEGD plasma.

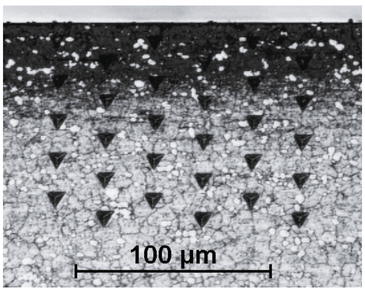


Fig. 6. Etched metallographic section of SW7M steel sample, the cross section of nitrided area after the microhardness measurement [10]

4. Active Screen Plasma Nitriding (ASPN)

Active Screen Plasma Nitriding (ASPN) is a synergistic combination of plasma nitriding methods in DCPN and post-discharge configurations. The scheme of the experimental set-up used for this nitriding method is shown in Fig. 7. Unlike the standard DC plasma nitriding in diode configuration, in the ASPN method plasma of abnormal glow discharge is formed on a mesh metal screen, called the active screen, surrounding the workpiece. The high potential of the cathode is connected to the active screen, while both the working table and nitrided elements are isolated. In the process, the workpiece together with the working table are placed on the floating potential or are biased by a small negative potential (BIAS potential). In the ASPN processes, the glow discharge is used to obtain the active particles and to heat the active screen, which then, due to radiation, heats up the nitrided workpiece. The ASPN method allows using the additional resistive heating system, which allows for independent control of the nitrided workpiece temperature and the power of the glow discharge. The plasma generated in the screen vicinity, is a mixture of electrons, ions and other active particles, which thanks to the use of an appropriate system of the process atmosphere circulation flow around the nitrided workpiece. The ions are also accelerated towards the surface of the workpiece thanks to the BIAS potential. Using this configuration, the device maintains the advantages of layers obtained by DCPN method without the possible drawbacks such as the hollow cathode effect or the edge effect [3, 5, 7, 13].

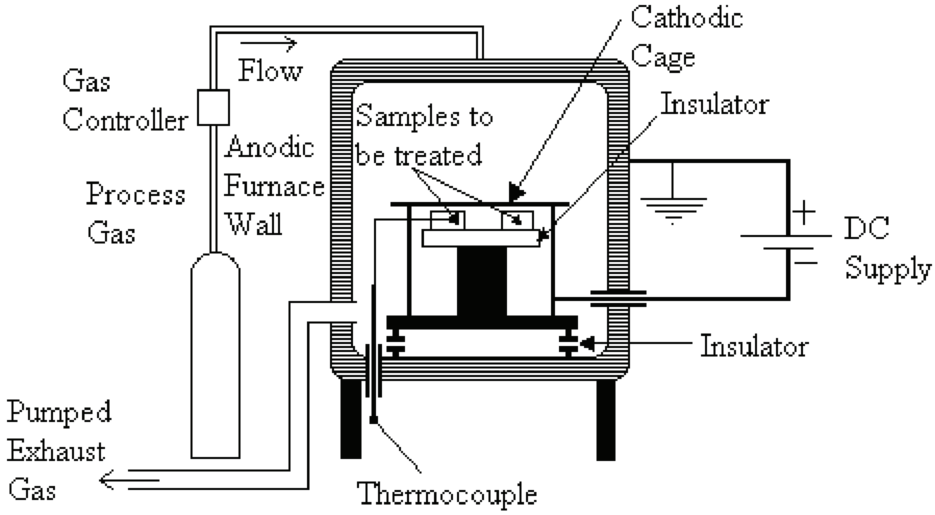


Fig. 7. Schematic illustration of the device used for ASPN method [3]

Nitriding in ASPN configuration is an innovative and relatively new technology, and therefore not fully understood by investigators also in terms of the occurring nitriding mechanisms. There are two fundamentally different and contradictory hypotheses about the basic mechanisms of plasma nitriding with the active screen, none of which is based on experimental irrefutable evidence. According to the first one, shown schematically in Fig. 8a, the main carriers of the nitrogen in the ASPN processes are the iron nitride particles sputtered from the active screen and transferred up to the workpiece surface. This hypothesis is based on SEEM and RD. analyses of the products of sputtering, which shows that these are submicron particles Fe_xN (x> 2) with a high surface area to volume ratio, which make them extremely active. On passing through the plasma atmosphere, the Fe_xN particles physically and chemically adsorb active nitrogen, and after the deposition on the workpiece surface, due to its high temperature, disintegrate and released nitrogen atoms diffuse into the nitrided elements, where some of them combine chemically to form iron nitrides. This mechanism is called a sputtering-adsorption-desorption model [3, 13-15].

The second hypothesis assumes that the main mechanism of nitrogen transport in the ASPN processes is bombardment by neutral particles. Nitrogen particles ionized near the polarized active screen are accelerated in its direction (Fig. 8b). If the ionized particle hits the active screen, it causes its sputtering. The sputtered cathode material may deposit on the workpiece surface (case A), and become a factor preventing the formation of the nitrided layer. If the ionized particles are accelerated through a hole in the screen, they move towards the workpiece surface, but they are hampered by the attraction of the high negative potential of the screen.

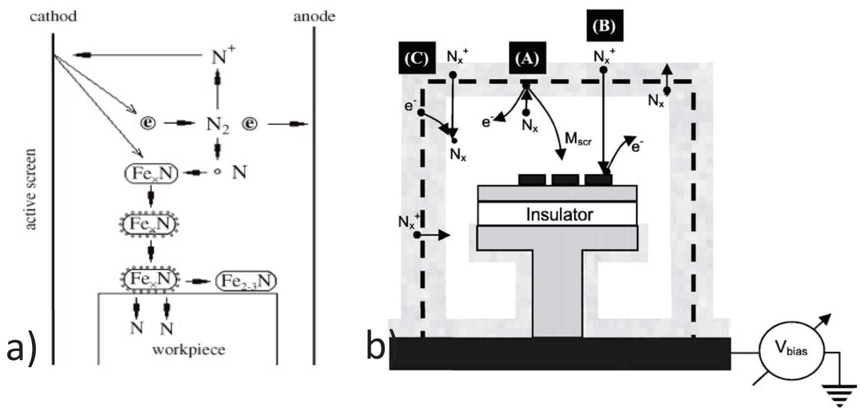
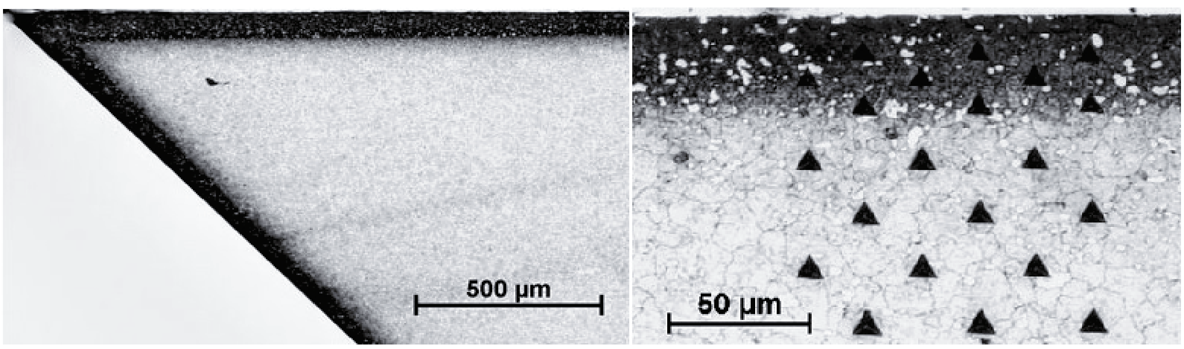


Fig. 8. Hypotheses about the transportation of nitrogen during active screen plasma nitriding process: a) „sputtering-adsorption-desorption" model [14], b) „neutral particle bombardment" model [15]

In this case, there is a possibility of their direct impact against the surface of the workpiece. This will happen only when the distance between the cathode and the workpiece is shorter than the range of the inhibited particles (case B). Some of the ionized particles, which pass through the hole in the screen may become neutral particles due to collisions with electrons. Once created, the fast neutral particles have a high kinetic energy and are not inhibited by the high potential of the screen. Thus, their range exceeds the range of the ions with the same energy, so that these particles bombard the surface of nitrided elements (case C). In this hypothesis, the bombardment of the workpiece by neutral particles is the main mechanism of nitrogen transport in ASPN processes. If, however, the distance between the active screen and the workpiece exceeds the range of the neutral particles, the major role in nitrogen transfer is taken over by the ionized particles attracted to the surface of the nitrided components by the BIAS potential [3, 6, 7, 14, 15].

Figure 9 shows the nitrided layer produced on the planning knife made from high-speed steel SW7M. The knife was nitrided by ASPN method. The image of the blade clearly shows that this method allows eliminating the drawback of edge effect that occurs in the DCPN method. In Fig. 8b, as in the case of metallographic samples presented previously, it can be observed that there is no surface area of hard nitrides ε.



a) *b)*

Fig. 9. Nitrided layer created on the planning knife [13]: a) cross-section of the planning knife after nitriding, b) microsection of the nitrided layer after measurement of the microhardness distribution

5. Conclusions

The article presents and briefly characterizes the three methods of plasma nitriding: Active Screen Plasma Nitriding (ASPN), Low Pressure Plasma Nitriding in AEGD plasma and Direct Current Plasma Nitriding (DCPN). Layers obtained by means of the discussed methods satisfy the basic requirements of the nitrided layers for special applications: high surface hardness, high resistance to abrasive wear, high resistance to fatigue and corrosion. The presented metallographic samples treated with the discussed methods show that it is possible to obtain a nitrided surface layer without hard but brittle zone of nitrides ε, which, if created, can adversely affect mechanical properties, and final dimensions of the nitrided elements. The possibility of strict control of operating parameters in each of the three discussed nitriding methods allows controlling the thickness, properties and phasing composition of nitrided layers obtained. The cited advantages show that the method of Direct Current Plasma Nitriding, Low Pressure Plasma Nitriding in AEGD plasma and Active Screen Plasma Nitriding can be used for nitriding of aircraft parts.

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